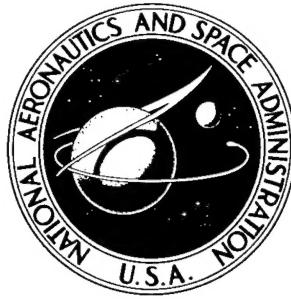


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EFFECT OF VARIATIONS IN  
SILICON AND IRON CONTENT  
ON EMBRITTLEMENT OF A  
COBALT-BASE ALLOY (L-605)

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by Gary D. Sandrock, Richard L. Ashbrook, and John C. Freche  
Lewis Research Center  
Cleveland, Ohio

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SUMMARY

An investigation was conducted to study the effect of variations in silicon and iron content in L-605 (HS-25) on room-temperature ductility and other mechanical properties after aging at 1600° F for various times up to 1000 hours. The silicon content of the alloy was investigated over a range from 0.12 to 1.00 percent, and the iron content over a range from 0.16 to 3.24 percent. These ranges are generally within the manufacturer's specified nominal composition limits for these elements. This investigation shows that for low silicon content heats (0.12 to 0.23 percent) the room-temperature ductility of L-605 sheet aged for 1000 hours at 1600° F was improved over that of the high silicon content heats (0.49 to 1.00 percent), as measured by tensile elongations. The former heats had elongations of 13 to 16 percent, the latter had elongations of 2 to 6 percent. Little apparent effect on room-temperature ductility after aging was observed as a result of the variation of iron content from 0.16 to 3.24 percent.

Aging at 1600° F reduced the room-temperature ultimate tensile strength of L-605 for all the compositions investigated, and no overall relation between silicon content and ultimate tensile strength after aging was observed. Low iron content heats generally had higher ultimate tensile strengths after aging for 1000 hours than did the high iron content heats. Hardness generally increased with aging time for all the heats, although this effect was less pronounced for the low silicon heats.

Upon aging, precipitates formed preferentially at grain and twin boundaries as well as randomly in the matrix. The lower silicon content heats had a lesser amount of precipitate after aging than did the high silicon content heats. Variations in iron content appeared to have little overall effect on microstructure after aging.

to page 3

INTRODUCTION

The cobalt-base alloy L-605 (HS-25) has many elevated-temperature uses. Its mechanical and physical properties are summarized in references 1 and 2. Because of its elevated-temperature strength, fabricability, and weldability

the alloy is of interest for aerospace applications. A potential application is its use in tubing and radiator components of advanced space power systems that must operate for mission times of thousands of hours. This alloy has a tendency, however, to become brittle after long-time exposure to high temperatures (ref. 3), a property obviously undesirable in engineering applications involving long-time exposure particularly those subject to mechanical and thermal cycling.

Jenkins (ref. 3) attributed the embrittlement of L-605 to the heavy precipitation of the intermetallic compound  $\text{Co}_2\text{W}$  during high-temperature exposure. Wlodek (ref. 4) suggested that the compound  $\text{Co}_2\text{W}$  is a stable Laves phase in this alloy. Because  $\text{Co}_2\text{W}$  is not an equilibrium phase in the Co-W binary system (ref. 5), Wlodek suggested that it is stabilized in L-605 by the silicon present. He contended that reduction in silicon content would lessen Laves phase precipitation and, in turn, reduce embrittlement. Some data supporting this contention are shown in reference 4 and the discussion of reference 4. It was also proposed in reference 4 that precipitation of carbides and the possible formation of hcp cobalt contributed to the embrittlement in L-605 and that the formation of hcp cobalt can be prevented by increasing the concentration of iron. Iron is believed to stabilize the fcc cobalt structure.

In view of the importance of retaining ductility in L-605 a program was undertaken at NASA Lewis Research Center to investigate the effect of wide variations of silicon and iron content, within the manufacturer's specifications, on mechanical properties of L-605 after aging. The room-temperature tensile strength and ductility, as well as the hardness of L-605, were determined after aging for various times up to 1000 hours at  $1600^{\circ}\text{ F}$ . Stress-rupture data were also obtained with the alloy in the unaged condition. In addition, metallurgical studies were made to obtain insight into the metallurgical mechanisms involved.

## EXPERIMENTAL PROCEDURE

### Material

Six special heats and two commercial heats, based on the manufacturer's standard practice prior to 1964 (see appendix) were obtained from the Union Carbide Corporation (Stellite Division) for use in this investigation. The manufacturer's commercial melting practice has recently been changed so as to achieve silicon contents similar to those provided in the low silicon special heats of this investigation. As wide a range of silicon and iron contents (generally within the manufacturer's nominal composition specifications) as was practically feasible at the time was obtained. The chemical compositions and grain sizes of the heats investigated are given in table I. Complete chemical analyses were determined by the supplier. The iron and silicon contents were also determined by an independent laboratory for all heats except heat 5. Silicon contents ranged from 0.12 to 1.00 weight percent, and iron contents ranged from 0.16 to 3.24 weight percent, as determined by the independent laboratory. These analyses are used in the data curves presented in this paper. Average ASTM grain size varied from 4 to 6.

TABLE I. - CHEMICAL COMPOSITIONS AND GRAIN SIZES OF HEATS INVESTIGATED

Heat number	Determined by independent laboratory		Determined by supplier										Average ASTM grain size
	Silicon	Iron	Silicon	Iron	Chromium	Tungsten	Carbon	Nickel	Manganese	Cobalt	Phosphorous	Sulfur	
1	0.23	0.57	0.20	0.60	20.02	15.02	0.13	10.40	1.52	Balance	0.001	0.019	6
2	.60	.49	.50	.58	20.16	15.42	.11	10.12	1.40	Balance	.001	.013	6
3	.73	.24	.96	.20	20.14	15.66	.11	10.20	1.44	Balance	.005	.008	5
b <sub>4</sub>	.55	1.60	.63	1.67	20.07	14.62	.08	10.06	1.52	Balance	.012	.011	4
b <sub>5</sub>	---	----	.49	1.85	20.09	15.05	.09	9.90	1.37	Balance	.014	.010	4
6	1.00	3.24	1.33, 1.12	2.90, 2.90	20.79	14.75	.10	10.14	1.11	Balance	.004	.010	6
7	.12	.16	0.04, 0.11	0.20, 0.23	20.41	14.32	.13	10.42	1.10	Balance	----	.012	6
8	.12	3.06	0.03, 0.07	2.85, 2.88	20.06	14.50	.12	10.27	1.10	Balance	----	.005	6
Manufacturer's specifications													
	a <sub>1</sub>	a <sub>3</sub>	19 to 21	14 to 16	0.05 to 0.15	9 to 11	1 to 2	Balance	-----	-----	-----	-----	

<sup>a</sup>Maximum according to manufacturer's specifications, ref. 1.<sup>b</sup>Manufacturer's standard practice prior to 1964.

All material was hot-rolled to approximately 0.050 inch sheet and mill-annealed (2250° F, rapid-air cooled) by the supplier.

#### Specimen Configuration

Figure 1 shows the tensile test specimen configuration used in this investigation. Machining was performed prior to aging.

#### Aging Procedure

The specimens were aged at 1600° F in air. This temperature was selected because the greatest rate of embrittlement was observed (ref. 4) with material that had been aged at this temperature. During aging, furnace temperature was continuously recorded. Specimen temperature was monitored periodically by means of a separate reference thermocouple located immediately adjacent to the specimens. Specimens

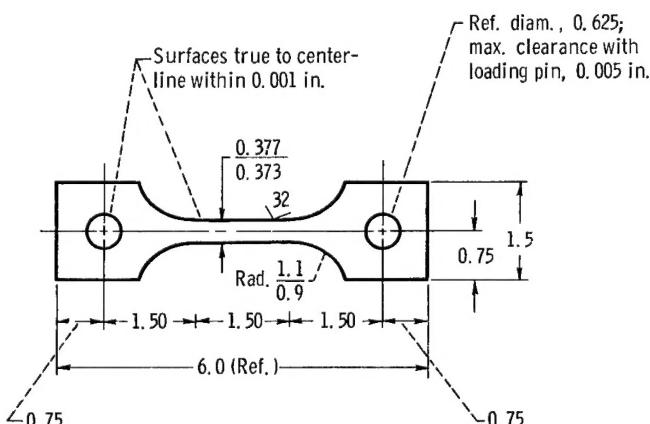


Figure 1. - Tensile test specimen. (Unless otherwise noted, dimensions may vary  $\pm 0.01$  in.)

from all heats were aged for 50, 200, and 1000 hours, then cooled in air to room temperature. Oxidation of the samples was slight, even after being exposed for 1000 hours at 1600° F.

#### Hardness Tests

Hardness testing was done on tensile specimens near the shoulder of the specimen outside of the test length. A standard Rockwell machine using the A scale (60 kg load, brale indentor) was employed. Prior to testing, any thin oxide film present was removed either by grinding or by using a low-pressure dental gritblasting unit.

#### Tensile tests

Tensile tests were made with a 60 000 pound capacity hydraulic tensile machine. All tests were performed at room temperature. A standard snap-on extensometer was used to obtain the load-strain curve to a strain of about 0.5 inch per inch. The extensometer was then removed and the test was continued to fracture. Strain rate was not directly controllable with this machine; however, an attempt was made to maintain a constant strain rate by conducting the tests at a constant valve setting. Minor variations in strain rate that might have occurred would not be expected to affect greatly the tensile properties of this alloy at room temperature (ref. 2). Elongation measurements were made over a 1-inch gage length. Yield strength was measured on the basis of 0.2-percent offset.

#### Metallographic studies

Longitudinal sections (in the rolling direction) of typical test specimens were taken for metallographic examination in the as-received (mill-annealed) condition and for each aging condition investigated. The as-received specimens were electrolytically etched in a hydrochloric acid - 0.1-percent hydrogen peroxide solution. Aged specimens were electrolytically etched in a boric acid-dilute sulfuric acid solution. After etching, specimens were swabbed with ammonia to remove stains. Photomicrographs were taken at magnifications of 750 and 250.

#### RESULTS AND DISCUSSION

The effects of wide variations in silicon and iron content (within the nominal composition range) on the mechanical properties and microstructure of L-605 after various aging times at 1600° F are discussed in the following sections. The tensile, hardness, and stress-rupture data are listed in tables II, III, and IV, respectively.

*Exposure  
time*

TABLE II. - SUMMARY OF TENSILE RESULTS

Heat number	Aging time at 1600° F, hr	Yield strength (0.2 - percent offset), psi	Ultimate tensile strength, psi	Elongation, percent	Heat number	Aging time at 1600° F, hr	Yield strength (0.2 - percent offset), psi	Ultimate tensile strength, psi	Elongation, percent
1	0	65 300 77 600	145 200 163 300	48.1 54.0	5	0	75 300 74 600 71 900	148 900 147 700 145 200	54.0 55.0 52.5
	50	69 700 66 700 64 600	141 400 141 200 140 400	19.2 27.0 27.0		55	74 500 76 300 75 600	133 000 134 400 136 400	12.0 10.7 12.3
	200	63 600 69 400 62 800	134 000 143 100 133 800	20.0 19.0 23.8		200	81 800 83 500 79 800	141 400 143 400 135 200	4.5 4.7 3.2
	1000	64 200 64 300 63 900	134 000 132 000 130 800	16.5 15.0 14.5		1000	78 600 80 400 79 800	124 400 127 700 127 100	2.0 2.0 1.7
2	0	70 600 71 300	153 700 153 600	45.0 40.2	6	0	90 100 89 600 97 000	166 900 166 200 167 700	47.4 47.0 46.9
	50	69 900 71 900 70 200	137 900 134 500 135 600	15.0 12.4 16.0		50	77 700 78 500 78 100	137 200 138 700 136 600	9.5 10.3 9.3
	200	71 900 70 200 72 500	130 500 129 800 140 400	4.5 7.4 9.4		200	81 100 76 500 79 300	133 500 132 800 135 100	3.3 3.7 4.2
	1000	72 100 71 700 72 600	131 900 130 700 133 300	5.5 5.2 7.2		1000	79 000 78 500 78 400	132 400 130 100 131 000	2.8 2.5 2.8
3	0	70 500 72 800	149 400 148 000	38.0 36.0	7	0	74 100 75 800 73 600	157 400 159 600 156 800	41.4 46.4 44.9
	50	77 500 80 300	141 000 140 900	8.0 7.0		50	75 500 74 400 75 700	148 400 142 800 148 100	25.9 21.9 25.0
	200	79 300 77 400 77 600	140 300 138 900 138 100	6.4 4.0 4.5		200	75 000 73 200 74 700	141 600 139 200 144 000	16.5 15.1 18.7
	1000	75 700 75 900 76 600	126 500 126 500 126 700	2.7 2.5 3.0		1000	75 800 68 000 71 600	144 800 127 800 135 600	14.7 11.0 12.2
4	0	70 600 71 000	138 500 142 500	45.0 42.2	8	0	69 300 69 900 67 900	151 100 148 400 148 600	51.4 49.3 46.4
	50	67 300 66 500	114 300 119 400	11.0 11.5		50	69 900 68 800 68 800	139 600 139 700 144 400	29.6 33.7 38.3
	200	70 700 71 200 70 900	128 000 125 800 128 300	6.9 5.5 7.4		200	66 000 65 800 66 000	138 200 139 000 138 200	30.7 34.6 29.2
	1000	73 400 71 600 70 600	119 100 120 200 119 400	3.2 3.5 2.7		1000	62 600 62 500 61 800	123 700 126 000 127 100	15.2 15.3 17.4

TABLE IV. - SUMMARY OF STRESS-TABLE III. - SUMMARY OF HARDNESS DATA

Heat number	Aging time at 1600° F, hr			
	0	50	200	1000
	Average hardness (Rockwell scale A)			
1	62.5	65.0	65.5	67.0
2	63.5	65.5	68.0	69.0
3	63.0	66.0	69.5	70.0
4	61.7	64.5	68.0	68.4
5	62.6	67.2	69.9	70.1
6	66.6	67.4	68.1	69.1
7	63.7	64.9	64.4	66.0
8	60.2	59.1	62.5	61.7

RUPTURE DATA

Heat number	Stress-rupture life at 1600° F and 17 500 psi, hr
1	57.7 71.4
2	69.9 65.2
3	63.4 51.4
4	44.2 32.4
6	34.6 43.5
7	30.9 25.8
8	17.3 18.7

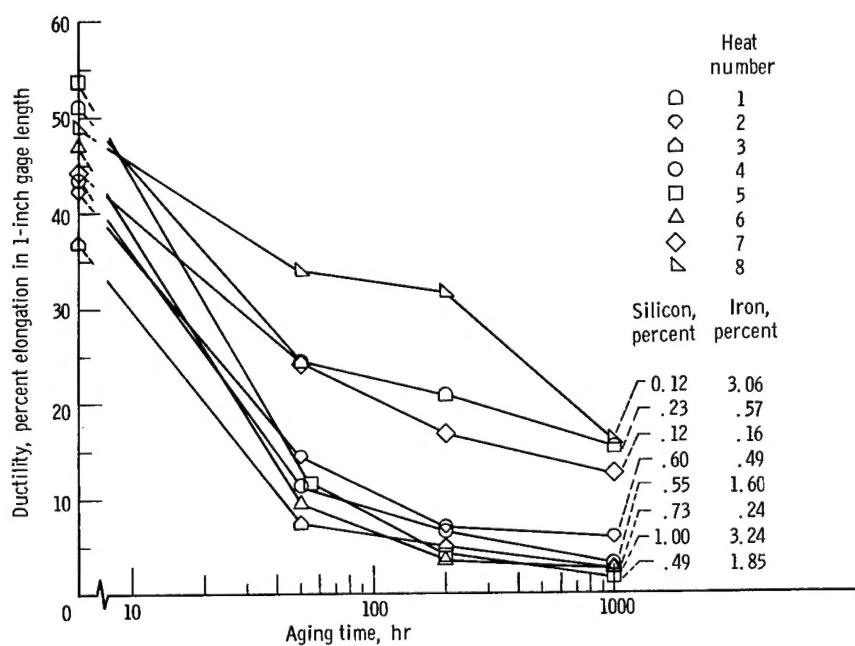


Figure 2. - Effect of aging time at 1600° F on average room temperature ductility.

## Mechanical Properties

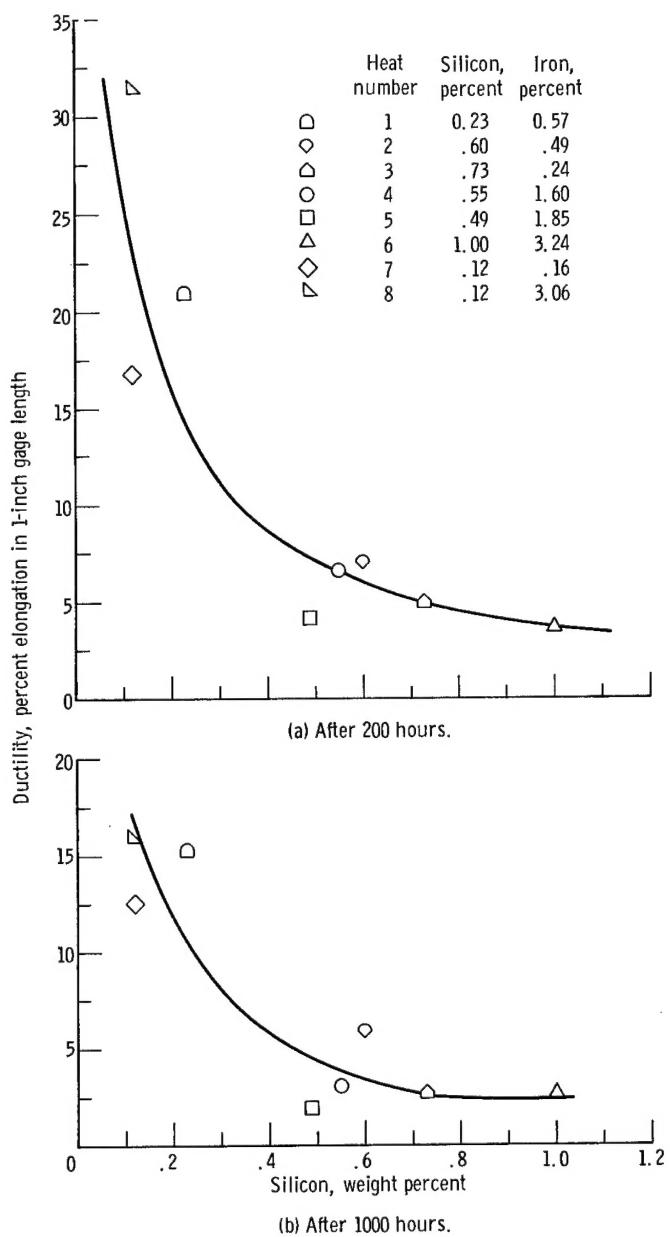


Figure 3. - Effect of silicon content on average room-temperature ductility after aging at 1600° F.

the lowest silicon content heats (7 and 8) always have the greatest ductility. Some crossover of the curves occurs so that the exact ranking of heats by ductility is not the same at each aging time. However, the trend toward improved ductility with lower silicon content is unmistakable, and a pronounced increase in ductility is clearly obtainable by reductions in silicon content to between 0.12 and 0.23 weight percent. This is illustrated more markedly in figure 3, which presents the relation between ductility and silicon content for all heats after aging for 200 and 1000 hours, respectively. Reducing the silicon content from 1 to 0.49 percent did not appear to have a pronounced effect on the ductility; however, further reductions in silicon content to 0.23 percent or less

Ductility. - The average room-temperature ductilities before aging and after aging at 1600° F for 50, 200, and 1000 hours are shown in figure 2. For purposes of comparison the heats may roughly be divided into two groups: high silicon content (0.49 to 1.00 percent) and low silicon content (0.12 and 0.23 percent). At all aging times the three heats with the low silicon contents had substantially greater tensile ductility than did the five high silicon content heats. After 50-hour aging treatments the low silicon content heats had elongations that ranged from approximately 24 to 34 percent, while the elongations of the high silicon content heats ranged between approximately 8 percent and 14 percent. After aging for 200 hours the low silicon content heats had elongations that ranged from approximately 17 to 32 percent as compared with only 3 to 7 percent for the high silicon content heats. After 1000-hour aging treatments the low silicon content heats still had considerably greater ductility, 13 to 16 percent as against 2 to 6 percent for the high silicon content heats. Four of the five high silicon content heats had elongations ranging between approximately 2 and 3 percent.

The highest silicon content heat (heat 6) did not always have the lowest ductility nor did both of

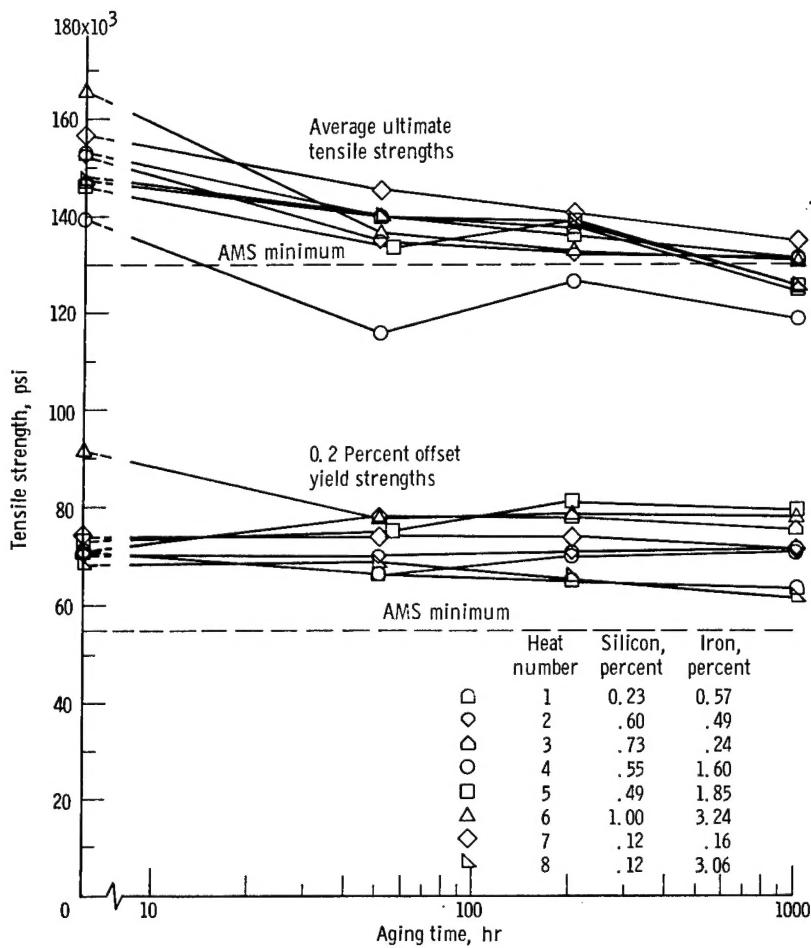


Figure 4. - Effect of aging time at 1600° F on average room-temperature tensile strength.

resulted in ductilities appreciably higher than those obtained with heats containing 0.49 to 1.00 percent silicon.

When heats within a narrow composition range of iron (0.16 to 0.57 percent) are considered, ductility after 200- and 1000-hour aging treatments is still seen to increase with decreasing silicon content (table II). On the other hand, when heats within narrow composition ranges of silicon (0.12 to 0.23 and 0.49 to 0.60 percent) are considered (fig. 2), no consistent trend of increasing ductility with increasing iron content is observed. In general, little apparent overall effect on postaging ductility was observed especially after 1000-hour aging treatments as a result of variations in iron content from 0.16 to 3.24 percent.

Tensile strength. - The average ultimate tensile strengths are plotted in figure 4 as a function of aging time at 1600° F for all heats investigated. In the as-received (mill-annealed) condition all eight heats investigated had ultimate tensile strengths greater than the 130 000-pound-per-square-inch minimum given by the Aerospace Materials Specification for L-605 (AMS 5537B, ref. 6). Interestingly, all six special heats investigated had ultimate tensile strengths

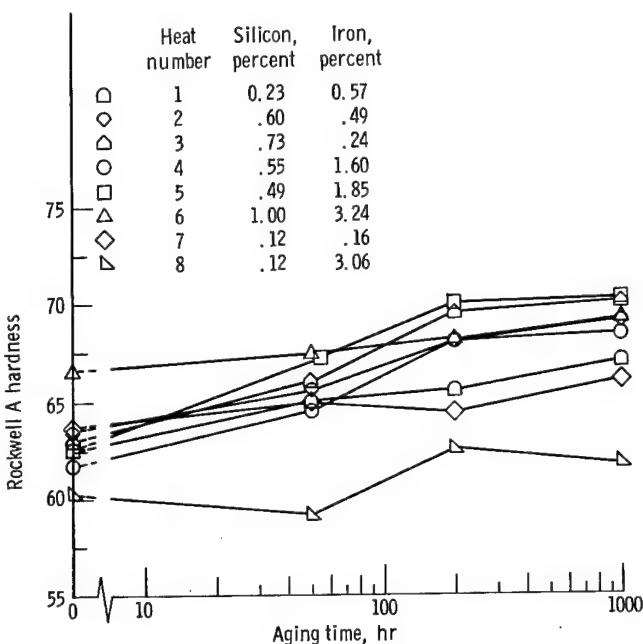


Figure 5. - Effect of aging time at 1600° F on average room-temperature hardness.

Because of the general loss in ultimate tensile strength with aging time, the ultimate tensile strengths of some heats fell below the AMS minimum of 130 000 pounds per square inch for L-605 after aging for 1000 hours. For all the aging times investigated, the tensile strength of heat 4 was less than the AMS minimum.

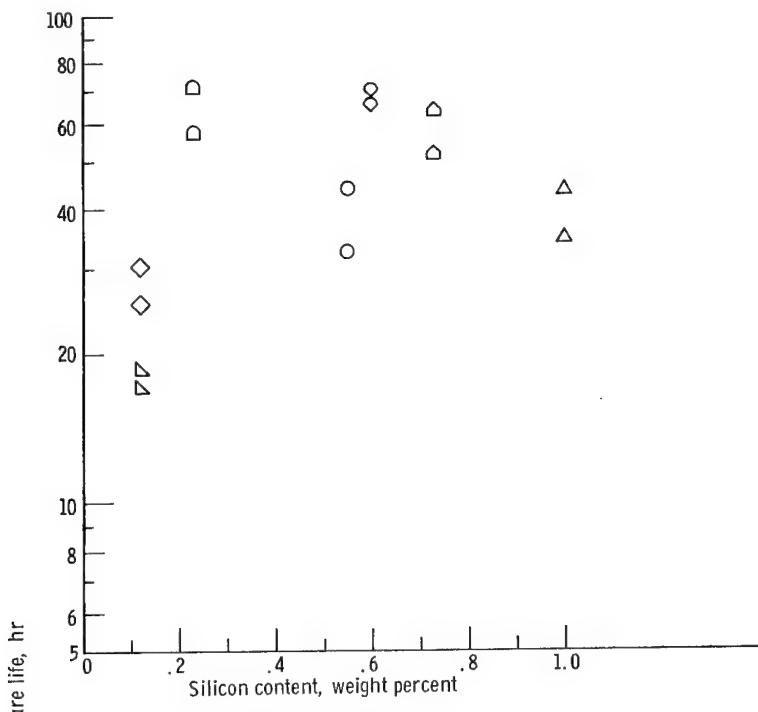
There is no clear-cut relation between silicon content and ultimate tensile strength after aging for 1000 hours; however, the low iron content heats generally had higher ultimate tensile strengths after aging for 1000 hours than did the high iron content heats. Interestingly enough after aging 1000 hours the tensile strengths of the low silicon content heats investigated were close to (either slightly above or below) the AMS specification minimum and generally superior to those of the two commercial heats.

Yield strength. - The 0.2-percent offset yield strengths are also plotted against aging time in figure 4. There is a general tendency for the high silicon content heats to have yield strengths higher than those of the low silicon content heats after aging. After aging for 50 hours, relatively little change in yield strength results with increased aging time for any of the heats. The highest silicon content heat (heat 6) had the highest yield strength in the mill-annealed condition. Its ultimate tensile strength (fig. 4) was similarly high in comparison with those of the other heats.

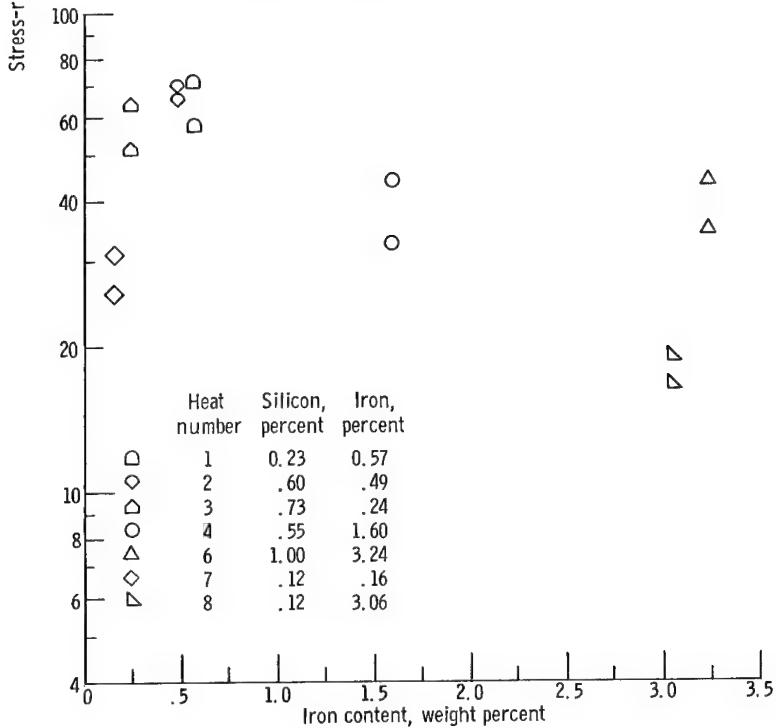
Hardness. - Figure 5 shows room-temperature hardness as a function of aging time for all the heats investigated. All data points indicated represent the average of five hardness readings. There is a general increase in hardness with aging time for all the heats. This increase in hardness is associated with

in the mill-annealed condition greater than the two commercial heats (heats 4 and 5). This indicates that the tensile strength of the mill-annealed sheet is not adversely affected by reductions in silicon content. Of course, some of the improvement in tensile strength may be due to the somewhat smaller grain size of the special heats (table I).

Room-temperature ultimate tensile strength generally decreased with aging time. The decrease was most pronounced after the first 50 hours. With aging times greater than 50 hours the decrease in ultimate tensile strength continued, but it was generally less marked. Heats 4 and 5 tended to regain part of their original strength after aging for 200 hours, but after aging for 1000 hours these two heats again showed a decrease in ultimate tensile strength.



(a) Effect of silicon.



(b) Effect of iron.

Figure 6. - Stress-rupture life of mill-annealed L-605 sheet at 1600° F and 17 500 pounds per square inch as a function of silicon and iron content.

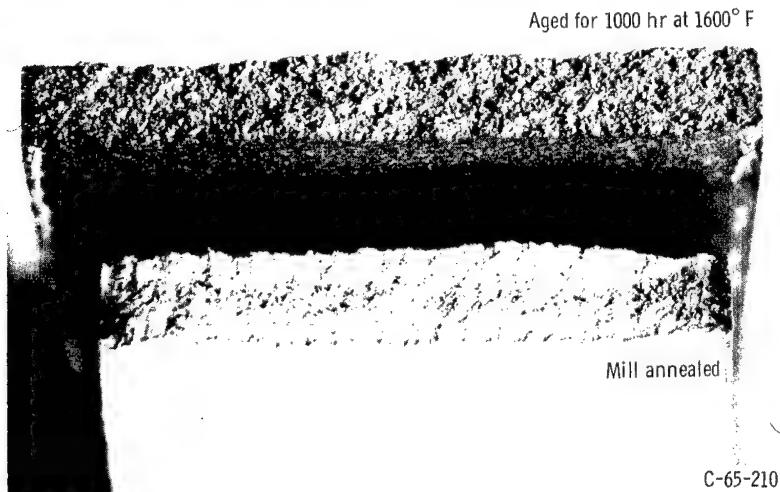


Figure 7. - Typical tensile fractures before and after aging (heat 3). X10.

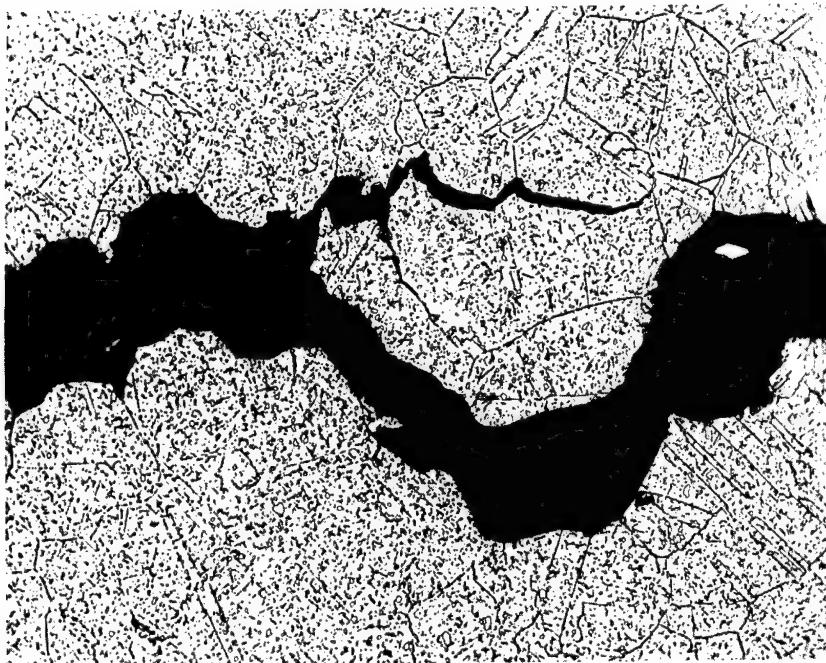
increased precipitation. In general, after aging, the hardness of the high silicon content heats increased more than that of the low silicon content heats. After aging for 1000 hours, for example, the three lowest silicon content heats (heats 1, 7, and 8) have the lowest hardnesses, Rockwell A hardness of 62 to 66 compared with 68 to 70 for the high silicon content heats. Metallographic studies (discussed in the section on the effect of silicon on microstructure, p. 14) show that substantially less precipitation occurs during aging in the low silicon content heats than occurs in the high silicon content heats. This fact would explain the lower hardness of the low silicon content heats and tend to substantiate the contention that Laves phase precipitation is reduced by lowered silicon content.

Stress-rupture. - Figure 6(a) shows the stress-rupture life at 1600° F and 17 500-pound-per-square-inch stress as a function of silicon content for all heats investigated in the unaged condition, except heat 5. No clear-cut relation between stress-rupture life and silicon content exists; however, the relatively low stress-rupture lives obtained with the two lowest silicon content heats suggest that the stress-rupture life may be adversely affected at very low silicon contents.

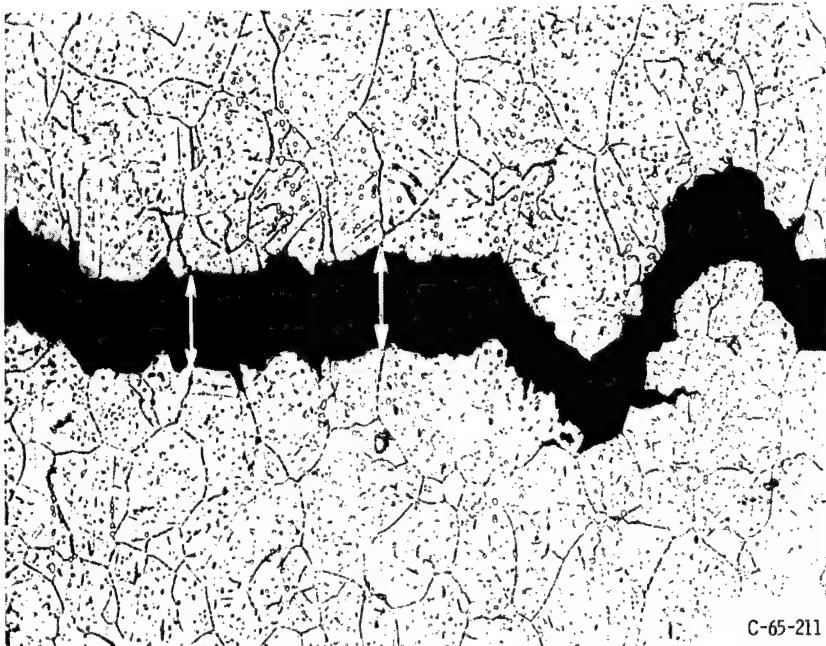
Figure 6(b) similarly shows the effect of iron content on stress-rupture life for the same test conditions. A trend toward decreasing stress-rupture life with increasing iron content seems to exist, although the trend is not well defined.

#### Fracture Modes

Visual observation of the fracture surfaces of specimens broken in tensile tests showed a different fracture mechanism occurring in specimens that have been aged as opposed to those that are in the mill-annealed condition. Figure 7 shows a typical example of this difference. The upper part of the figure shows the fracture surface of a tensile specimen from heat 3 aged for 1000 hours at 1600° F. A jagged fracture surface is evident. The lower part of the figure shows the fracture surface of a mill-annealed, or unaged, specimen from the



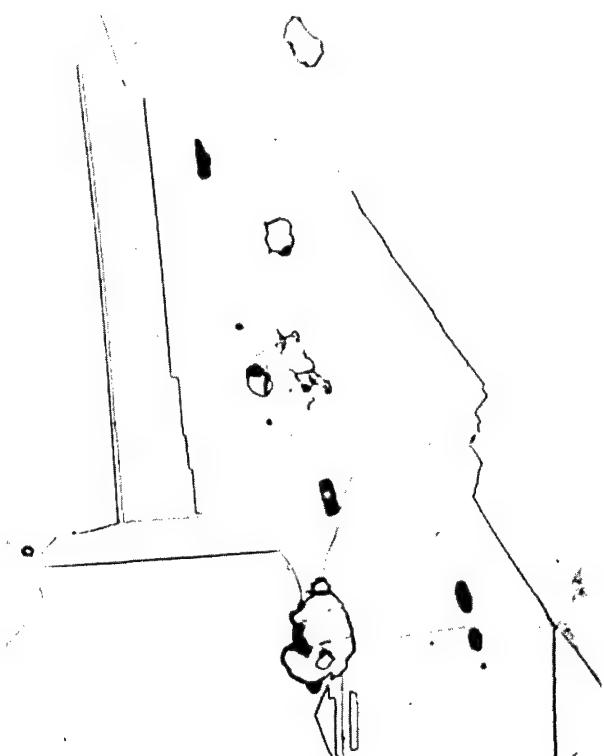
(a) 0.73 Percent silicon (heat 3).



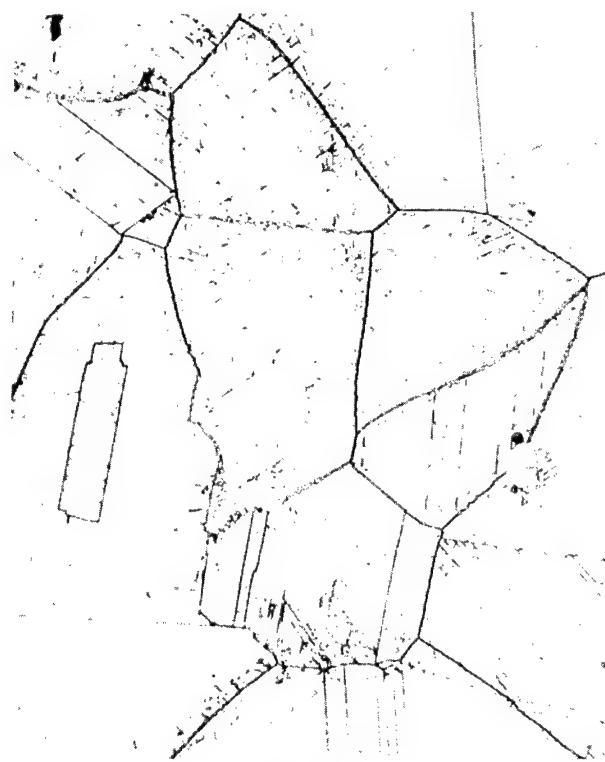
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(b) 0.12 Percent silicon (heat 7).

Figure 8. - Effect of silicon content on tensile fracture of L-605 sheet aged for 1000 hours at 1600° F. X250.  
(Arrows indicate some grain boundaries that can be traced across fracture.)



(a) 0 Hours.



(b) 50 Hours.



(c) 200 Hours.



(d) 1000 Hours.

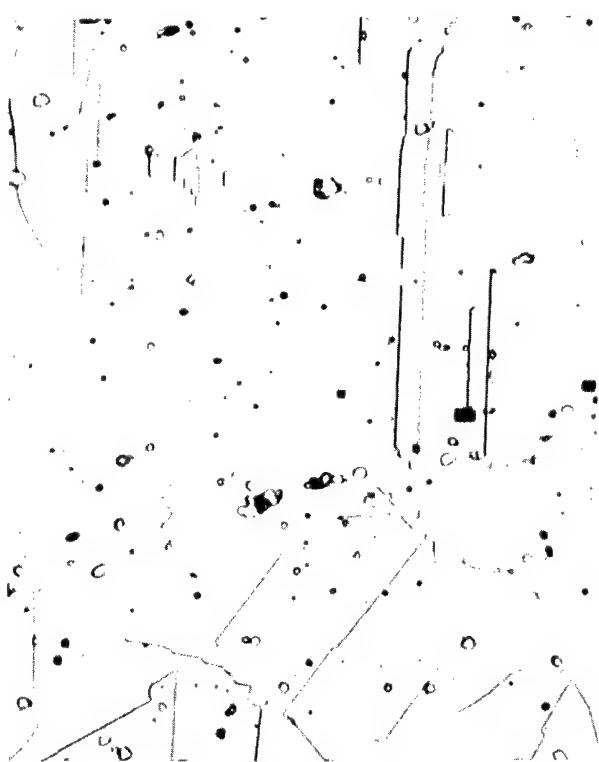
Figure 9. - Effect of aging time at 1600° F on microstructure of intermediate silicon and iron content heat (0.55 percent Si - 1.60 percent Fe (heat 4)). X750.

same heat. The surface is fibrous in nature, and fracture appears to be typical of the shear failures encountered in ductile materials. The fracture surface in the aged specimen was at 90° to the tensile axis while the fracture surface in the mill-annealed specimen was at an angle of about 60° to the tensile axis. Another aspect to be noted in the figure is the difference in width of each section. A much greater reduction in area occurred with the more ductile mill-annealed specimen. The types of fractures shown in figure 7 are generally typical of those encountered in all heats in the mill-annealed and aged conditions.

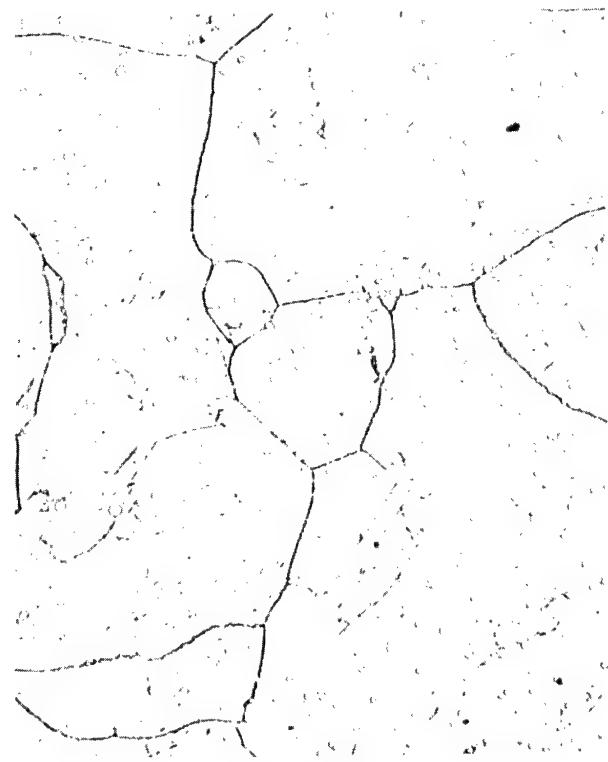
It was observed at higher magnifications, however, that the amount of silicon had some influence on the fracture mode of specimens after aging. Figure 8(a) shows the fracture of a tensile bar of heat 3 aged 1000 hours (same conditions as those in fig. 7, top) at a magnification of 250. Heat 3 is a high silicon content heat (0.73 percent Si) that had an elongation of only 2.7 percent after 1000-hour aging. Fracture was almost entirely intergranular, apparently because of the heavy precipitation particularly along grain boundaries. Figure 8(b) shows a typical tensile fracture incurred in a specimen of heat 7, a low silicon content heat (0.12 percent Si) that had an average elongation of 12.6 percent after 1000-hour aging. In this case, although some intergranular fracture occurred, a substantial degree of transgranular fracture took place as well. Grain boundaries can be traced across the fracture as indicated by the arrows in figure 8(b). Substantially less precipitation occurred in the specimen from heat 7. The amount of precipitate, that in turn is affected by silicon content, thus appears to have a noticeable effect on the nature of the fracture encountered.

#### Effects of Iron and Silicon Contents on Microstructure After Aging

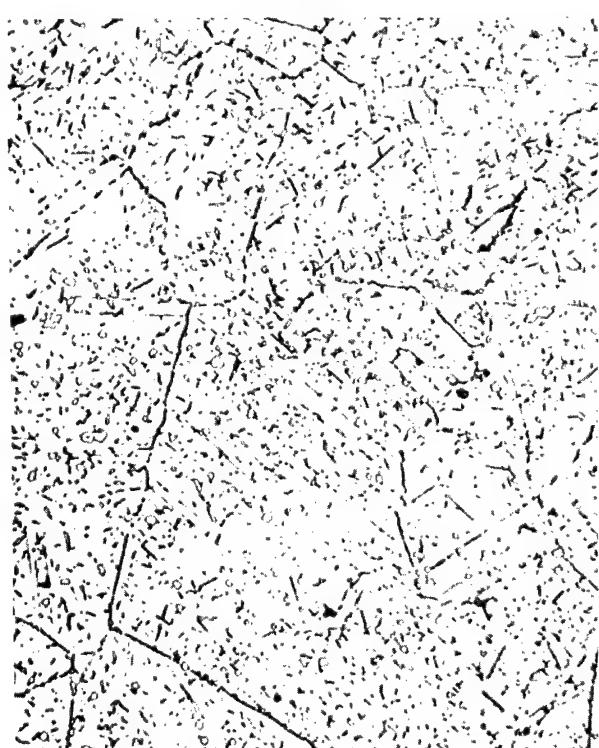
Intermediate silicon and iron content. - Figure 9 shows the microstructure of a commercial heat of L-605 (heat 4) that was made by the manufacturer's standard practice for this alloy prior to 1964. Figure 9(a) shows the as-received (mill-annealed) material. The microstructure consists largely of a solid solution of fcc cobalt. Twinning is evident in the grains. A few carbides that were not taken into solution during the annealing treatment are scattered throughout the matrix. After aging for 50 hours at 1600° F, precipitation is evident (fig. 9(b)). Initial nucleation is particularly pronounced along the grain and twin boundaries, although some also occurs randomly within the grains. After aging for 200 hours the amount of precipitate, both in the grain boundaries and within the grains, was greatly increased (fig. 9(c)). It should also be noted that many of the randomly located precipitate particles are rodlike or platelike in shape and tend to be lined up in preferred crystallographic directions. After aging for 1000 hours, the precipitate particles grew and possibly additional precipitate particles were formed (fig. 9(d)). The microstructure is similar to that observed (ref. 4) by Wlodek who identified the Laves phase ( $\text{Co}_2\text{W}$ ) in aged heats of similar silicon content. The structure shown in figure 9(d) is quite brittle; only about 3.1 percent tensile elongation was measured at fracture with tensile specimens aged for 1000 hours at 1600° F.



(a) 0 Hours.



(b) 50 Hours.



(c) 200 Hours.



(d) 1000 Hours.

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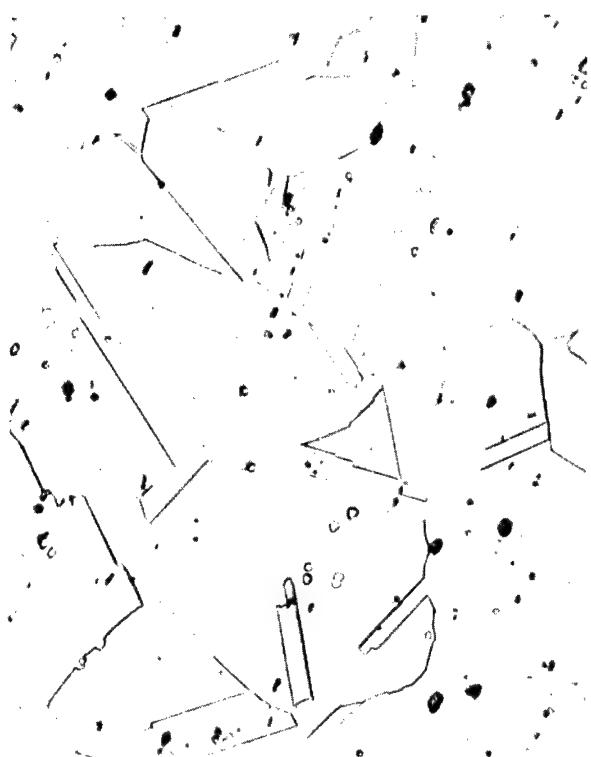
Figure 10. - Effect of aging time at 1600° F on microstructure of high silicon - low iron content heat (0.73 percent Si - 0.24 percent Fe (heat 3)). X750.

High silicon - low iron content. - Figure 10 shows the effect of aging on the microstructure of heat 3, which is high in silicon content (0.73 percent Si) and low in iron content (0.24 percent Fe). The microstructure of the as-received material for heat 3 (fig. 10(a)) is similar to that of the standard practice heat 4 (fig. 9(a)) except for the slightly smaller grain size and the larger amount of retained carbides. With aging, precipitation occurs in a manner similar to that already described for the intermediate silicon and iron content heat (heat 4). Figure 10(b) shows the microstructure of heat 3 after aging for 50 hours. Precipitation, as in heat 4, was pronounced along the grain boundaries. Somewhat more precipitation took place, however, in the higher silicon content heat, especially within the grains. This may be a contributing factor to the somewhat higher tensile strength and hardness obtained for heat 3 (see figs. 4 and 5) in this condition. After aging for 200 hours (fig. 10(c)) the grains were well filled with precipitate particles. After 1000 hours (fig. 10(d)), some particle growth could be seen. A structure similar to that obtained in the intermediate silicon and iron content heat (fig. 9(d)) resulted. This similarity in structure probably accounts for the fact that the postaging ductilities for both heats are almost the same (fig. 2).

Low silicon - high iron content. - Figure 11 shows the effect of aging on the microstructure of heat 8, which is a low silicon - high iron content heat (0.12 percent Si and 3.06 percent Fe). This composition resulted in the best ductility after aging, regardless of aging time, of all the heats investigated (fig. 2). It also had the lowest hardness. The microstructure (fig. 11) shows a substantially lower amount of precipitate at all aging times than occurred for either the intermediate silicon and iron content or the high silicon - low iron content heat. This smaller amount of precipitate could account for both the higher ductility and the lower hardness obtained after all aging times with the low silicon - high iron content heat. Figures 11(b), (c), and (d) show the microstructure after 50-, 200-, and 1000-hour aging treatments, respectively. Precipitation occurs primarily along the grain and twin boundaries as well as randomly within the grains, as was also noted in the intermediate silicon and iron and the high silicon - low iron content heats. It is evident that the major difference lies in the amount of precipitate.

Low silicon - low iron content. - Figure 12 shows the effect of aging on the microstructure of heat 7, which is low in both silicon and iron contents (0.12 percent Si and 0.16 percent Fe). Both the low silicon - high iron content heat considered in the previous section and the low silicon - low iron content heat have the same silicon content (0.12 percent Si); however, the latter heat has a much lower iron content (0.16 percent Fe). Comparing these two compositions (figs. 11 and 12) shows the effect of iron content on microstructure. In general, the microstructures are very similar. Thus, iron does not appear to have a pronounced effect on the amount and nature of the precipitate, at least at low silicon contents. The ductility of the low silicon - low iron content heat (heat 7) was substantially less at intermediate aging times than that of the low silicon - high iron content heat (heat 8). The reason for this difference is not apparent from a comparison of the microstructures (figs. 11 and 12).

Effect of silicon on amount of precipitate after 1000-hour aging. - The effect of silicon content has already been illustrated insofar as ductility is concerned. The microstructural effects have been discussed to a limited extent in the preceding subsections. To better observe the effect of silicon content



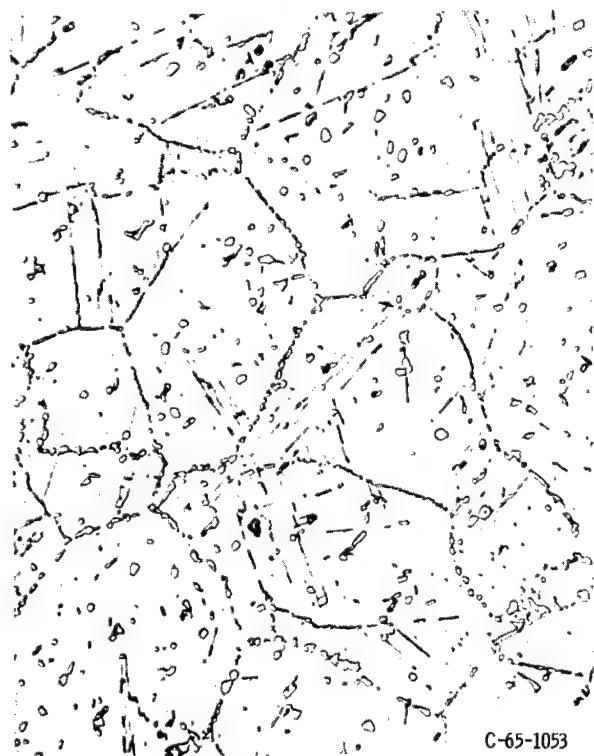
(a) 0 Hours.



(b) 50 Hours.



(c) 200 Hours.



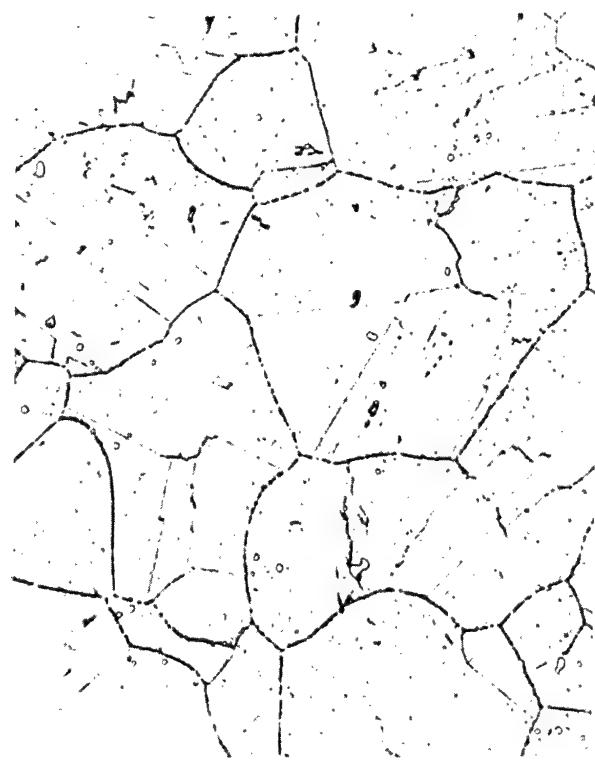
(d) 1000 Hours.

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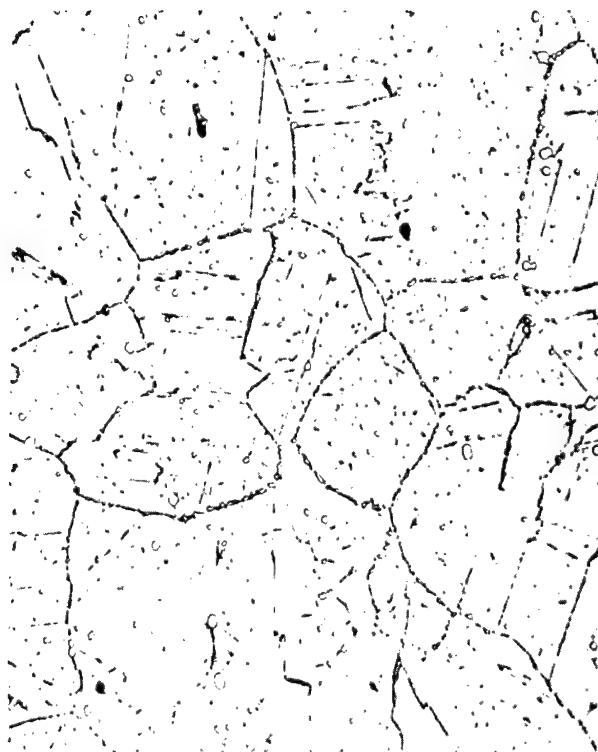
Figure 11. - Effect of aging time at 1600° F on microstructure of low silicon - high iron content heat (0.12 percent Si - 3.06 percent Fe, (heat 8). X750.



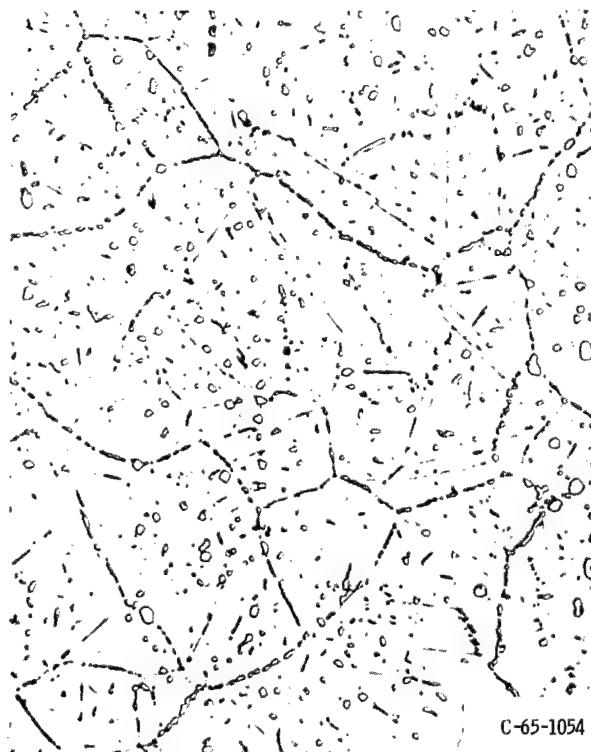
(a) 0 Hours.



(b) 50 Hours.



(c) 200 Hours.



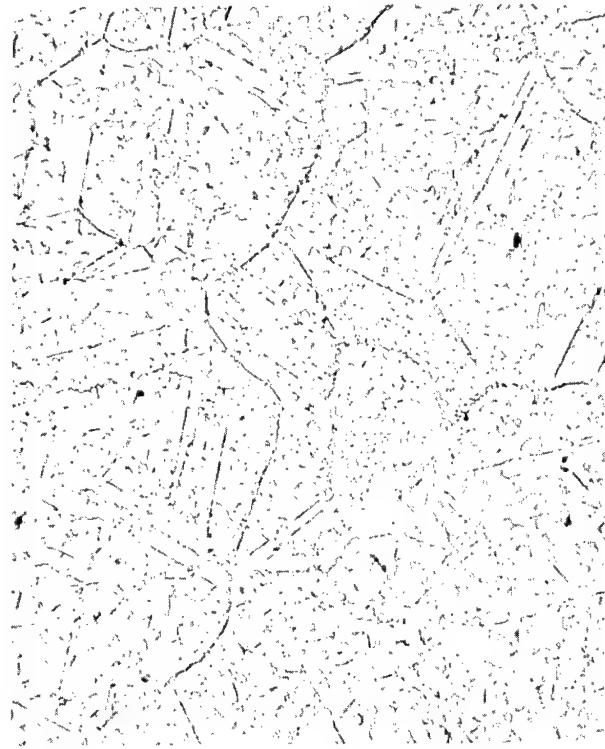
(d) 1000 Hours.

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Figure 12. - Effect of aging time at 1600° F on microstructure of low silicon - low iron content heat (0.12 percent Si - 0.16 percent Fe (heat 7)). X750.



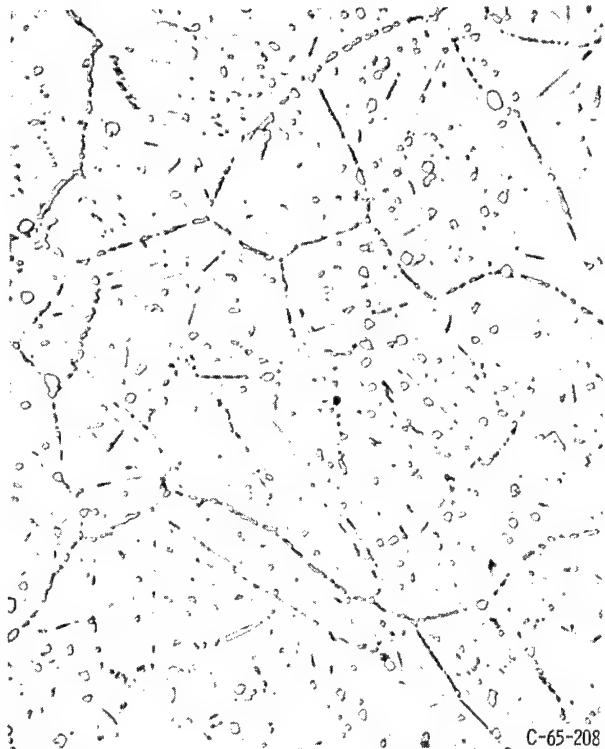
(a) 0.73 Percent silicon (heat 3).



(b) 0.60 Percent silicon (heat 2).



(c) 0.23 Percent silicon (heat 1).



(d) 0.12 Percent silicon (heat 7).

Figure 13. - Effect of silicon content on microstructure of L-605 aged for 1000 hours at 1600° F; iron content for heats ranged from 0.16 to 0.57 percent. X750.

on the microstructure of L-605, photomicrographs are compared in figure 13 for heats of varying silicon content (0.12 to 0.73 percent) but relatively constant iron content (0.16 to 0.57 percent) after aging for 1000 hours. The two highest silicon content heats, 0.73 and 0.60 percent silicon, respectively, (figs. 13(a) and (b)) both show heavy precipitation. With a reduction to 0.23 percent silicon (fig. 13(c)), however, a definite decrease in the amount of precipitate is evident. Figure 13(d) shows the microstructure of the lowest silicon heat (0.12 percent). The amount of precipitate is less than that in any of the other heats. These metallographic studies show that a reduction in silicon content greatly reduces precipitation after aging. This reduction in the amount of precipitate is believed to be associated with increased ductility in this alloy after aging.

X-ray diffraction analysis of the 0.23 and 0.60 percent silicon heats (heats 1 and 2) conducted by Wlodek (discussion, ref. 4) indicated only traces of Co<sub>2</sub>W (Laves phase) in the lower silicon heat and substantially greater amounts in the higher silicon heat. Although precipitation of the Laves phase was greatly reduced in the lower silicon content heats, appreciable quantities of precipitates were still present after aging. Many of these precipitate particles are probably carbides of the M<sub>6</sub>C type as identified by Wlodek in the 0.23 percent silicon content heat (discussion of ref. 4). The presence of such carbides particularly along grain boundaries could contribute to the degree of embrittlement that still exists, even in the low silicon content heats.

#### SUMMARY OF RESULTS

The following results were obtained from an investigation to determine the effect of silicon and iron contents on the room-temperature ductility and other mechanical properties of L-605 after aging at 1600° F for various times up to 1000 hours:

1. Reductions in silicon content increased ductility for all aging times (50, 200, and 1000 hr). For example, after aging for 1000 hours at 1600° F, the tensile elongations for the low silicon content heats (0.12 to 0.23 percent) ranged from approximately 13 to 16 percent as compared with 2 to 6 percent for the high silicon content heats (0.49 to 1.00 percent).
2. Little apparent overall effect on postaging ductility was observed as a result of variations of iron content from 0.16 to 3.24 percent.
3. The ultimate tensile strength of all the heats generally decreased with aging time. There was no overall relation between silicon content and ultimate tensile strength after aging. The low iron content heats generally had higher ultimate tensile strengths than those of the high iron content heats after aging for 1000 hours.
4. Hardness generally increased with aging time for all the heats investigated. In general, the hardness of the high silicon content heats increased more than the hardness of the low silicon content heats. For example, after aging for 1000 hours at 1600° F, the low silicon content heats had Rockwell A

hardnesses ranging from 62 to 66 compared with hardnesses of 68 to 70 for the high silicon content heats.

5. The fracture surface in as-received (mill-annealed) specimens had a fibrous appearance typical of that observed in ductile materials; whereas the fracture surface of aged specimens, regardless of silicon or iron content, was jagged in nature. In the aged condition fracture in the high silicon heats was predominantly intergranular, whereas in low silicon heats there was a substantial degree of transgranular fracture as well.

6. Aging at 1600° F resulted in pronounced precipitation both preferentially along grain and twin boundaries and randomly throughout the matrix. The lower silicon content heats had a lesser amount of precipitate after aging than did the high silicon content heats. Variations in iron content appeared to have little effect on the microstructure after aging.

Lewis Research Center,  
National Aeronautics and Space Administration,  
Cleveland, Ohio, May 25, 1965.

## APPENDIX

NEW MELTING PRACTICE

Recently the Union Carbide Corporation (Stellite Division) introduced a new melting practice into the production of L-605 (HS-25), which can provide very low silicon contents. Data obtained by the manufacturer (private communication from D. W. Schulz, Union Carbide Corp.) for heats made by this practice are given in table V. These data show that low silicon content results in elongations of 6.4 to 17 percent after 1000-hour aging treatments at 1600° F. It should be noted that elongations of 2 to 3 percent were obtained with similarly aged specimens from heats 4 and 5 of the present investigation. The latter heats were made by the manufacturer's standard practice prior to 1964. The tensile strengths for the heats listed in table V are somewhat lower than those obtained in the present investigation with low silicon content heats.

In summary, it appears that improved postaging ductility in L-605 sheet can also be obtained in commercial practice by melting procedures that provide a low silicon content. end

TABLE V. - PRELIMINARY TENSILE DATA FOR L-605 SHEET  
MADE BY NEW MELTING PRACTICE<sup>a</sup>

Heat number	Composition			Yield strength <sup>b</sup> (0.2-percent offset), psi	Ultimate tensile strength, psi	Elongation <sup>b</sup> , percent
	Silicon	Carbon	Iron			
<sup>c</sup> 64-590	0.02	0.10	1.86	67 000	123 000	16
<sup>c</sup> 64-627	.03	.04	1.98	67 000	121 000	17
<sup>d</sup> L4-1677S	.04	.09	1.52	65 000	118 000	11
<sup>d</sup> L4-1696S	.10	.12	2.27	61 000	108 000	6.4

<sup>a</sup>Private communication from D. W. Schulz, Union Carbide Corporation.

<sup>b</sup>Aged 1000 hr at 1600° F, average of 2 to 4 tests.

<sup>c</sup>Laboratory heats.

<sup>d</sup>Production heats.

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